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MEMORANDUM

DATE 12 January 1999

TO David Bennett, WAM, U S. EPA, Region X

FROM: Michelle Turner, Chemist, WESTON, Seattle
Roger McGinnis, Senior Environmental Chemist, WESTON, Seattle

SUBJECT. Validation of Organotin Data
Laboratory Batch: K9805975
Site Duwamish River

WORK ASSIGNMENT NO 46-35-0JZZ

WORK ORDER NO 4000-019-038-5200-00

DOC. CONTROL NO 4000-019-038-AAAK

cc: Bruce Woods, RAP-WAM, U.S EPA, Region X
Dena Hughes, Site Manager, WESTON, Seattle (memo only)
Kevin Mundell-Jackson, Database Management, WESTON

The quality assurance review of twelve sediment samples, laboratory batch K9805975, collected from the Duwamish River has been completed The sediment samples were analyzed for organotins by Columbia Analytical Services of Kelso, Washington. Samples were analyzed by gas chromatography with an FPD detector. The samples were numbered:

98364003	98364011	98364012	98364024	98364025
98364026	98364027	98364028	98364029	98364030
98364031	98364032			

Data Qualifications

The following comments refer to the laboratory performance in meeting the quality control criteria described in the technical specifications of the laboratory subcontract The review follows the format described in the *National Functional Guidelines for Organic Data Review*

(EPA OSWER Directive 9240.1, February 1994), modified to include specific requirements of analytical methods

1 Timeliness

Holding time limits of 7 days for sample extraction and additional 7 days for analysis were established in the project Sampling and Analysis plan. All samples were extracted 9 days after sample collection, exceeding the 7 day holding time criteria. However, prior to extraction, samples were stored frozen, thus extending the holding time. Samples were extracted within the 12 month holding time recommended by PSEP for frozen samples

All samples met extraction holding time criteria but exceeded analysis holding time criteria as follows:

Sample ID	Date Collected	Date Extracted	Date Analyzed	No of Days
98364003	8/31/98	9/9/98	9/17/98	8 days (analysis)
98364011	8/31/98	9/9/98	9/17/98	8 days (analysis)
98364012	8/31/98	9/9/98	9/17/98	8 days (analysis)
98364024	9/1/98	9/9/98	9/18/98	9 days (analysis)
98364028	9/1/98	9/29/98	10/7/98	8 days (analysis)

Sample results and detection limits were qualified as estimated (UJ/J).

2 Detection Limits

Detection limits met project required quantitation limits with the following exceptions

Sample	Compound	QL Goal (µg/Kg)	Reported QL (µg/Kg)
98364003	n-Butyltin	10	55
98364024	n-Butyltin	10	85

Where quantitation limit goals were exceeded, undetected analytes were qualified (UI) to indicate matrix interference

3. Initial Calibration

A seven-point initial calibration was performed prior to each analytical batch. The percent relative standard deviation for the initial calibration was within limits of less than 25 percent RSD.

4. Continuing Calibrations

Continuing calibration check was performed after every 10 samples. Target analytes were within required limits for the continuing calibrations with the percent difference for a mid-range standard less than 25 percent.

5 Blanks

a) *Laboratory Method Blanks*

Laboratory method blank frequency criteria were met. No target analytes were reported in laboratory method blanks.

b) Field Blanks

No field blanks were associated with this SDG.

6. Surrogate Compound Recovery

Surrogate recovery goals for Tripropyltin were established in the project Sampling and Analysis Plan at 60 to 130 percent for sediment Based on conversations with the laboratory an additional surrogate, Tripentyltin was added and historical laboratory control chart limits were also used for data qualification. Laboratory limits are presented below.

Surrogate Compound	Sediment Limits
Tripropyltin	20 - 195%
Tripentyltin	20 - 172%

Surrogate compound percent recoveries exceeded the QC limits for the following samples:

Sample	Surrogate	Percent Recovery
98364012	Tripentyltin	59
98364024	Tripentyltin	138
98364026	Tripropyltin	56
98364028	Tripropyltin	55
98364029	Tripropyltin	49
98364030	Tripropyltin	52
98364032	Tripropyltin	53
98364028MS	Tripropyltin	56
98364026	Tripentyltin	56
98364027	Tripentyltin	58
98364028	Tripentyltin	59
98364029	Tripentyltin	47
98364030	Tripentyltin	43
98364031	Tripentyltin	41
98364032	Tripentyltin	56
K980929-LCS	Tripentyltin	47
K980929-MB	Tripentyltin	47

Samples results and detection limits were qualified as estimated (UJ/J) when both surrogate recoveries were outside the QC limits.

7. Laboratory Control Sample (LCS)

LCS recovery goals for Butyltins were established in the project Sampling and Analysis Plan at 60 to 130% for sediment. Based on conversations with the laboratory, historical control chart limits of 20 to 164 percent for sediment were also used for data qualification.

Laboratory control sample percent recoveries met QC guidelines (P-project, L-laboratory), with the following exceptions:

LCS	Analyte	Percent Recovery	QC Limit	Associated Samples
K980909-LCS	n-Butyltin	34	60-130 (P) 20-164 (L)	98364003 98364011 98364012 98364024
K980909-DLCS	n-Butyltin	24	60-130 (P) 20-164 (L)	98364003 98364011 98364012 98364024
K980929-LCS	Tetrabutyltin	16	60-130 (P) 20-164 (L)	98364025 through 98364032
K980929-LCS	Dibutyltin	59	60-130 (P) 20-164 (L)	98364025 through 98364032
K980929-LCS	n-Butyltin	16	60-130 (P) 20-164 (L)	98364025 through 98364032

Sample results were qualified as estimated (J) when LCS recoveries were outside project limits. Undetected results were qualified as estimated (UJ) when LCS recoveries were outside project limits.

8. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

The following matrix spike recovery goals were established in the project Sampling and Analysis Plan at for sediment

Analyte	% Recovery
Tributyltin	40 - 120%
Dibutyltin	30 - 120%
n-Butyltin	10 - 120%

All MS/MSD sample percent recoveries and relative percent differences (RPDs) met QC guidelines

9 Field Duplicate Analysis

Samples 98364011 and 98364012 were submitted to the laboratory as "blind" field duplicates Relative percent differences (RPDs) were less than 35 percent for sediment samples with the following exceptions

Analyte	RPD
Tributyltin	45 5

Results for analytes listed above were qualified as estimated (J) in samples 98364011 and 98364012.

10 Sample Analysis

A cursory review of raw data was performed. Deliverables were accurate and complete A duplicate analysis was performed on sample 98364025, RPD values were less than 35 percent for analytes with concentrations greater than five times the reporting limit The case narrative indicated that the LCS recovery of Tetrabutyltin extracted on 9/29/98 was outside the laboratory QC limits. As the MS and DMS were within QC limits, no action was taken. No other problems were noted in the case narrative

11. Laboratory Contact

No laboratory contact was required.

Data Assessment

Upon consideration of the data qualifications noted above, the data are ACCEPTABLE for use except where flagged with data qualifiers that modify the usefulness of the individual values.

Data Qualifiers

U - The compound was analyzed for, but was not detected

UJ - The compound was analyzed for, but was not detected The associated quantitation limit is an estimate because quality control criteria were not met

J - The analyte was positively identified, but the associated numerical value is an estimated quantity because quality control criteria were not met or because concentrations reported are less than the quantitation limit or lowest calibration standard.

R - Quality control indicates that data are unusable (compound may or may not be present). Resampling and reanalysis are necessary for verification.

N - Presumptive evidence of presence of material (tentative identification).

I - Elevated reporting limit due to matrix interference.

COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client:	Roy F Weston, Inc	**Service Request:**	K9805975
Project:	Duwamish River/4000-027-001-2019-38	**Date Collected:**	8/31/98
Sample Matrix:	Sediment	**Date Received:**	9/1/98

Butyltins

Sample Name	98364003	Units	ug/Kg (ppb)
Lab Code	K9805975-002	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	9/9/98	9/17/98	ND UJ	
Tri-n-butyltin	Method	Butyltins	5	5	9/9/98	9/17/98	34 J	
Di-n-butyltin	Method	Butyltins	5	5	9/9/98	9/17/98	ND UJ	
n-Butyltin	Method	Butyltins	55	5	9/9/98	9/17/98	ND UJI	B

B	The MRL is elevated because of matrix interferences
D	The MRL is elevated because of matrix interferences and because the sample required diluting

MGT 1/26/99

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Analytical Report

Client:	Roy F Weston, Inc	**Service Request:**	K9805975
Project:	Duwamish River/4000-027-001-2019-38	**Date Collected:**	8/31/98
Sample Matrix:	Sediment	**Date Received:**	9/1/98

Butyltins

Sample Name	98364011	Units	ug/Kg (ppb)
Lab Code	K9805975-010	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	9/9/98	9/17/98	ND UJ	
Tri-n-butyltin	Method	Butyltins	5	5	9/9/98	9/17/98	170 J	
Di-n-butyltin	Method	Butyltins	5	5	9/9/98	9/17/98	52	
n-Butyltin	Method	Butyltins	5	5	9/9/98	9/17/98	27	

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MQT 1/2/99

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Analytical Report

Client:	Roy F Weston, Inc	**Service Request:**	K9805975
Project:	Duwamish River/4000-027-001-2019-38	**Date Collected:**	8/31/98
Sample Matrix:	Sediment	**Date Received:**	9/1/98

Butyltins

Sample Name	98364012	Units	ug/Kg (ppb)
Lab Code	K9805975-011	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	9/9/98	9/17/98	7	J
Tri-n-butyltin	Method	Butyltins	5	5	9/9/98	9/17/98	270	
Di-n-butyltin	Method	Butyltins	5	5	9/9/98	9/17/98	59	
n-Butyltin	Method	Butyltins	5	5	9/9/98	9/17/98	30	

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Analytical Report

Client:	Roy F Weston, Inc	**Service Request:**	K9805975
Project:	Duwamish River/4000-027-001-2019-38	**Date Collected:**	9/1/98
Sample Matrix:	Sediment	**Date Received:**	9/2/98

Butyltins

Sample Name	98364024	Units	ug/Kg (ppb)
Lab Code	K9805975-012	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	9/9/98	9/18/98	ND UJ	
Tri-n-butyltin	Method	Butyltins	5	5	9/9/98	9/18/98	29 J	
Di-n-butyltin	Method	Butyltins	5	5	9/9/98	9/18/98	11 J	
n-Butyltin	Method	Butyltins	85	5	9/9/98	9/18/98	ND UJ I	B

B	The MRL is elevated because of matrix interferences
D	The MRL is elevated because of matrix interferences and because the sample required diluting

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Analytical Report

Client:	Roy F Weston, Inc	**Service Request:**	K9805975
Project:	Duwamish River/4000-027-001-2019-38	**Date Collected:**	9/1/98
Sample Matrix:	Sediment	**Date Received:**	9/2/98

Butyltins

Sample Name	98364025	Units	ug/Kg (ppb)
Lab Code	K9805975-013	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	ND UJ	
Tri-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	40	
Di-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	20 J	
n-Butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	8 J	

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Analytical Report

Client:	Roy F Weston, Inc	**Service Request:**	K9805975
Project:	Duwamish River/4000-027-001-2019-38	**Date Collected:**	9/1/98
Sample Matrix:	Sediment	**Date Received:**	9/2/98

Butyltins

Sample Name	98364026	Units	ug/Kg (ppb)
Lab Code	K9805975-014	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	ND UJ	
Tri-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	9 J	
Di-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	ND UJ	
n-Butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	6 J	

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Analytical Report

Client:	Roy F Weston, Inc	**Service Request:**	K9805975
Project:	Duwamish River/4000-027-001-2019-38	**Date Collected:**	9/1/98
Sample Matrix:	Sediment	**Date Received:**	9/2/98

Butyltins

Sample Name	98364027	Units	ug/Kg (ppb)
Lab Code	K9805975-015	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	ND UJ	
Tri-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	19	
Di-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	ND UJ	
n-Butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	8 J	

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Analytical Report

Client:	Roy F Weston, Inc	**Service Request:**	K9805975
Project:	Duwamish River/4000-027-001-2019-38	**Date Collected:**	9/1/98
Sample Matrix:	Sediment	**Date Received:**	9/2/98

Butyltins

Sample Name	98364028	Units	ug/Kg (ppb)
Lab Code	K9805975-016	Basis	Dry
Test Notes			

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	1	1	9/29/98	10/7/98	ND	UJ
Tri-n-butyltin	Method	Butyltins	1	1	9/29/98	10/7/98	ND	
Di-n-butyltin	Method	Butyltins	1	1	9/29/98	10/7/98	ND	
n-Butyltin	Method	Butyltins	1	1	9/29/98	10/7/98	ND	

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Analytical Report

Client:	Roy F Weston, Inc	**Service Request:**	K9805975
Project:	Duwamish River/4000-027-001-2019-38	**Date Collected:**	9/1/98
Sample Matrix:	Sediment	**Date Received:**	9/2/98

Butyltins

Sample Name	98364029	Units	ug/Kg (ppb)
Lab Code	K9805975-017	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	ND UJ	
Tri-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	77 J	
Di-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	26	
n-Butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	17	

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Analytical Report

Client:	Roy F Weston, Inc	**Service Request:**	K9805975
Project:	Duwamish River/4000-027-001-2019-38	**Date Collected:**	9/2/98
Sample Matrix:	Sediment	**Date Received:**	9/3/98

Butyltins

Sample Name	98364030	Units	ug/Kg (ppb)
Lab Code	K9805975-018	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	6	5	9/29/98	10/6/98	ND UJ	B
Tri-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	250 J	
Di-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	24	
n-Butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	55	

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D	The MRL is elevated because of matrix interferences and because the sample required diluting

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Analytical Report

Client:	Roy F Weston, Inc	**Service Request:**	K9805975
Project:	Duwamish River/4000-027-001-2019-38	**Date Collected:**	9/2/98
Sample Matrix:	Sediment	**Date Received:**	9/3/98

Butyltins

Sample Name	98364031	Units	ug/Kg (ppb)
Lab Code	K9805975-019	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	ND UJ	
Tri-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	100	
Di-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	ND UJ	
n-Butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	67 J	

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Analytical Report

Client:	Roy F Weston, Inc	**Service Request:**	K9805975
Project:	Duwamish River/4000-027-001-2019-38	**Date Collected:**	9/2/98
Sample Matrix:	Sediment	**Date Received:**	9/3/98

Butyltins

Sample Name	98364032	Units	ug/Kg (ppb)
Lab Code	K9805975-020	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	ND UJ	
Tri-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	39 J	
Di-n-butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	14	
n-Butyltin	Method	Butyltins	5	5	9/29/98	10/6/98	11	

D The MRL is elevated because of matrix interferences and because the sample required diluting

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Analytical Report

Client:	Roy F Weston, Inc	**Service Request:**	K9805975
Project:	Duwamish River/4000-027-001-2019-38	**Date Collected:**	NA
Sample Matrix:	Sediment	**Date Received:**	NA

Butyltins

Sample Name	Method Blank	Units	ug/Kg (ppb)
Lab Code	K980909-MB	Basis	Dry
Test Notes			

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	1	1	9/9/98	9/16/98	ND	
Tri-n-butyltin	Method	Butyltins	1	1	9/9/98	9/16/98	ND	
Di-n-butyltin	Method	Butyltins	1	1	9/9/98	9/16/98	ND	
n-Butyltin	Method	Butyltins	1	1	9/9/98	9/16/98	ND	

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